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Evaluation of Parts-per-Billion (PPB) Organic Cylinder Gases for Use as Audits during Hazardous Waste Trial Burn Tests

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EVALUATION OF PARTS-PER-BILLION (PPB) ORGANIC CYLINDER GASES FOR USE AS AUDITS DURING HAZARDOUS WASTE TRIAL BURN TESTS

by

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16. ABSTRACT

A repository of 14 gaseous organic compounds at parts-per-billion levels (7-430 ppb) in compressed gas cylinders has been established by the Environmental Protection Agency. This repository was established to provide audit materials for use in conducting performance audits to assess the accuracy and precision of Principal Organic Hazardous Constituent (POHC) measurements, especially those performed during hazardous waste trial burn tests. Five- and nine-component mixtures in a balance gas of nitrogen were prepared in aluminum cylinders for use in the performance audit program. The five-component gas cylinders contain carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene. The nine-component mixtures contain trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane, dichlorodifluoromethane, bromoethane, methyl ethyl ketone and 1,1,1-trichloroethane.

Studies of all 14 gaseous organic compounds were performed to determine the stability of the compounds and the feasibility of using them in performance audits. Results indicate that all of the 14 compounds were adequately stable to be used as reliable audit materials. Subsequent to completion of the stability studies, several performance audits were conducted using the repository gases to assess the accuracy and precision of the volatile organic sampling-train (VOST) and bag measurement methods. The audit results indicate that laboratories can use these methods to analyze POHC with accuracy to usually within ± 50% of the audit concentrations.

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EVALUATION OF PARTS-PER-BILLION (PPB) ORGANIC CYLINDER GASES FOR USE AS AUDITS DURING HAZARDOUS WASTE TRIAL BURN TESTS

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A repository of 14 gaseous organic compounds at parts-per-billion levels (7 - 430 ppb) in compressed gas cylinders has been established by the Environmental Protection Agency. This repository was established to provide audit materials for use in conducting performance audits to assess the accuracy and precision of Principal Organic Hazardous Constituent (POHC) measurements, especially those performed during hazardous waste trial burn tests. Five and nine component mixtures in a balance gas of nitrogen were prepared in aluminum cylinders for use in the performance audit program. The five-component gas cylinders contain carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene. The nine-component mixtures contain trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane, dichlorodifluoromethane, bromoethane, methyl ethyl ketone and 1,1,1-trichloroethane.

Studies of all 14 gaseous organic compounds were performed to determine the stability of the compounds and the feasibility of using them in performance audits. Results indicate that all of the 14 compounds were adequately stable to be used as reliable audit materials. Also, the estimation of specific uncertainties associated with the analysis of these audit materials and how those uncertainties are used to assign a total uncertainty to the final analyte concentration for five organics is described.

Subsequent to completion of the stability studies, several performance audits were conducted using the repository gases to assess the accuracy and precision of the volatile organic sampling-train (VOST) and bag measurement methods. The audit results indicate that laboratories can use these methods to analyze POHC with accuracy to usually within $\pm$ 50 percent of the audit concentrations.

Introduction

During the past few years, the need for accurate standards for the determination of Principle Organic Hazardous Constitutents (POHC) in trace levels (<50 parts-per-billion levels) during Hazardous Waste Trial burn tests has become increasingly evident. The ability to correlate data among analytical laboratories depends, to a large extent, on the uncertainty in the concentration of the standards used in the analyses. The scientific and regulatory problems associated with toxic organic pollution cannot be answered satisfactorily without adequately characterized standards.

The Quality Assurance Division of EPA's Environmental Monitoring Systems Laboratory has initiated a program to supply audit standards for use during hazardous waste trial burn tests. The Research Triangle Institute (RTI), under contract to the U.S. Environmental Protection Agency (EPA), has responded to this need through development of a repository of 14 gaseous compounds containing POHC at parts-per-billion (ppb) levels in compressed gas cylinders. The main objectives of this program are: (1) to provide accurate gas mixtures to assess the relative accuracy of source measurement systems during hazardous waste trial burn tests; (2) to verify the manufacturer's certified analysis of the multi-component gas mixture; and (3) to determine the stability of the POHC in gas cylinders.

The repository currently contains 14 compounds selected on the basis of anticipated needs of EPA's Office of Solid Waste. A list of these 14 compounds is shown in Table I. The gaseous compounds in Table I were purchased in compressed gas cylinders from commercial suppliers. The balance gas for all gas mixtures is nitrogen, and the cylinder construction material is aluminum. The stabilities of the compounds in the repository are determined over time from the measurement of cylinder concentrations calibrated with permeation tube standards. A description of the analytical system, the estimation of specific and net uncertainties associated with the measurement process, stabilities of these compounds in the compressed gas cylinder and how these cylinder gases are successfully used for performance audits to assess and document the accuracy of POHC measurements are described in this paper.

Experimental Procedures

Analysis of the cylinder gases is required to verify the concentrations reported by the company which prepared the gas mixtures and also to measure concentration changes with time, that is, to estimate the stability of the compounds. The contents of each cylinder are initially analyzed at RTI when they are first received from the manufacturer and are then analyzed periodically over the course of a year to evaluate the stability of the mixtures. All measurements are made by gas chromatography (GC).

Instrumentation

Analyses of the audit cylinder gases at RTI are performed with a Hewlett-Packard Model 5880A gas chromatograph with flame ionization and electron capture detectors. The electron capture detector (ECD) has been used principally for measurement of all the chlorinated hydrocarbons except vinyl

Table I. PPB level organic gases in repository.

Group	No. of Cylinders	Concentration Range of Each Compound (ppb)
Group I*	20	7-90
	12	90-430
Group II**	13	7-90
	3	90-430

*Group I Compounds - carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene.

**Group II Compounds - trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane, dichlorodifluoromethane, bromoethane, methyl ethyl ketone, and 1,1,1-trichloroethane

chloride. Vinyl chloride, benzene, methyl ethyl ketone and acetonitrile are analyzed with a flame ionization detector (FID). The gas chromatographic parameters used in the measurement of individual compounds are listed in Table II. A schematic of the gas chromatographic system is shown in Figure 1.

Gaseous samples are injected onto the columns by means of a six-port gas sampling valve constructed of Hastalloy C mounted near the injection port. The valve is equipped with interchangeable, stainless steel sample loops to allow the injection of a fixed volume of gas. Sample loops of 1 cm^3 and 10 cm^3 are used for ECD and FID, respectively.

Standardization and Measurement

Calibration of the gas chromatograph involves measurement of known concentrations of gases in nitrogen. A custom-designed permeation system (Figure 2) which produces known concentrations of organics in nitrogen is used for calibration. Permeation tubes purchased from commercial suppliers are used as calibration standards for all 14 organic compounds. A permeation tube for each organic is placed in a temperature-controlled chamber (30° $\pm$ 0.1°C) and pure nitrogen is passed over all the permeation tubes at a known flow rate using Tylan mass flow controllers. The resultant gaseous mixture is further diluted, if necessary, by addition of nitrogen in the mixing chamber. The final mixture is then drawn into the GC sample injection valve by a small pump downstream. The permeation tubes are not used until their permeation rates, which are determined periodically by measured weight loss, have been well established. The permeation rate of each permeation tube is determined every 15 to 30 days by weight-loss determination of each tube. Permeation rates are checked both before and after analysis and the average of the last three values were used for rate determination.

In order to avoid calibration errors associated with non-linear detectors (for example, ECD), at least two calibration standards are prepared each time a sample is analyzed. Both standards are prepared usually within 20 percent of the expected sample concentrations. For analysis using FID, standard concentrations are matched to sample concentrations at least within a factor of 2 and are within 20 percent at concentrations below 70 ppb. Two to six calibration standards are analyzed on each day of sample analysis. Also, in order to insure injection precision over the entire day, the calibration standards are injected both at the beginning and end of each day to measure the calibration system stability. All analyses data reduction are performed via the HP 5880 integrator and software.

Sample Analysis Procedure

The procedures used for cylinder gas analysis are as follows: Initially the sample loops are purged with cylinder gas sample at approximately 27 cc/min. for 4 minutes before injection. Whenever the 10 cc and not the 1 cc loop is used, it is purged with the cylinder gas for an additional 2 minutes before injection onto the column. Sample gas flow is controlled by slowly opening the needle valve attached to the tank. After purging the loop, the gas flow is shut off at the needle valve. Four seconds after a flowmeter registers zero flow through the sample loop, injection is made manually onto

Table II. GC Conditions for Analysis of PPB-Level Repository Gases

Compounds	Detector	Injector Temp.	Column Temp./ Carrier Gas	Column Type	Retention Time (min.)	Comments
			GROUP I			
Vinyl chloride Benzene	FID at 300°C 35 cc/min Hydrogen 500 cc/min Air or 500 cc/min 37% O_2 in Nitrogen	80°C	80°C, Isothermal/ Nitrogen at 55 cc/min	10 ft. x 1/8" stainless steel column packed with 20% SP 2100 w/0.1% Carbowax 1500, 100/120 mesh	0.7 3.5	10 cc sample loop used; 37% O_2 in N_2 as Detector oxidant added to increase sensitivity below 50 ppb
Chloroform Carbon tetrachloride Perchloroethylene	ECD at 330°C	110°C	100°C, Isothermal/ Argon w/5% Methane; at 40 cc/min	10 ft. x 1/8" stainless steel column packed with 20% SP 2100 w/0.1% Carbowax 1500, 100/120 mesh	1.6 2.1 3.9	1.0 cc sample loop used
			GROUP II			
Freon 12 Freon 11 Bromomethane Trichloroethylene 1,2-Dibromoethane	ECD at 330°C	110°C	80°C, Isothermal/ Argon w/5% Methane at 35 cc/min	10 ft. x 1/8" stainless steel column packed with 20% SP 2100 w/0.1% Carbowax 1500, 100/120 mesh	0.6 1.1 0.9 2.4 6.2	1 cc sample loop used
1,2-Dichloroethane 1,1,1-Trichloroethane	ECD at 330°C	110°C	110°C, Isothermal/ Argon w/5% Methane at 35 cc/min	20 ft. x 1/8" stainless steel column packed with 10% SP 1000, 80/100 Supelcoport	5.2 2.8	1 cc sample loop used
Acetonitrile and Methyl Ethyl Ketone	FID at 300°C 35 cc/min Hydrogen 500 cc/min Air or 500 cc/min 37% O_2 in Nitrogen	180°C	170°C, Isothermal/ Nitrogen at 35 cc/min	6 ft. x 1/8" stainless steel column packed with Porapak Q, 60/80 mesh	1.6 4.0	10 cc sample loop used; 37% O_2/N_2 used below 50 ppb; conditioned column 3 days

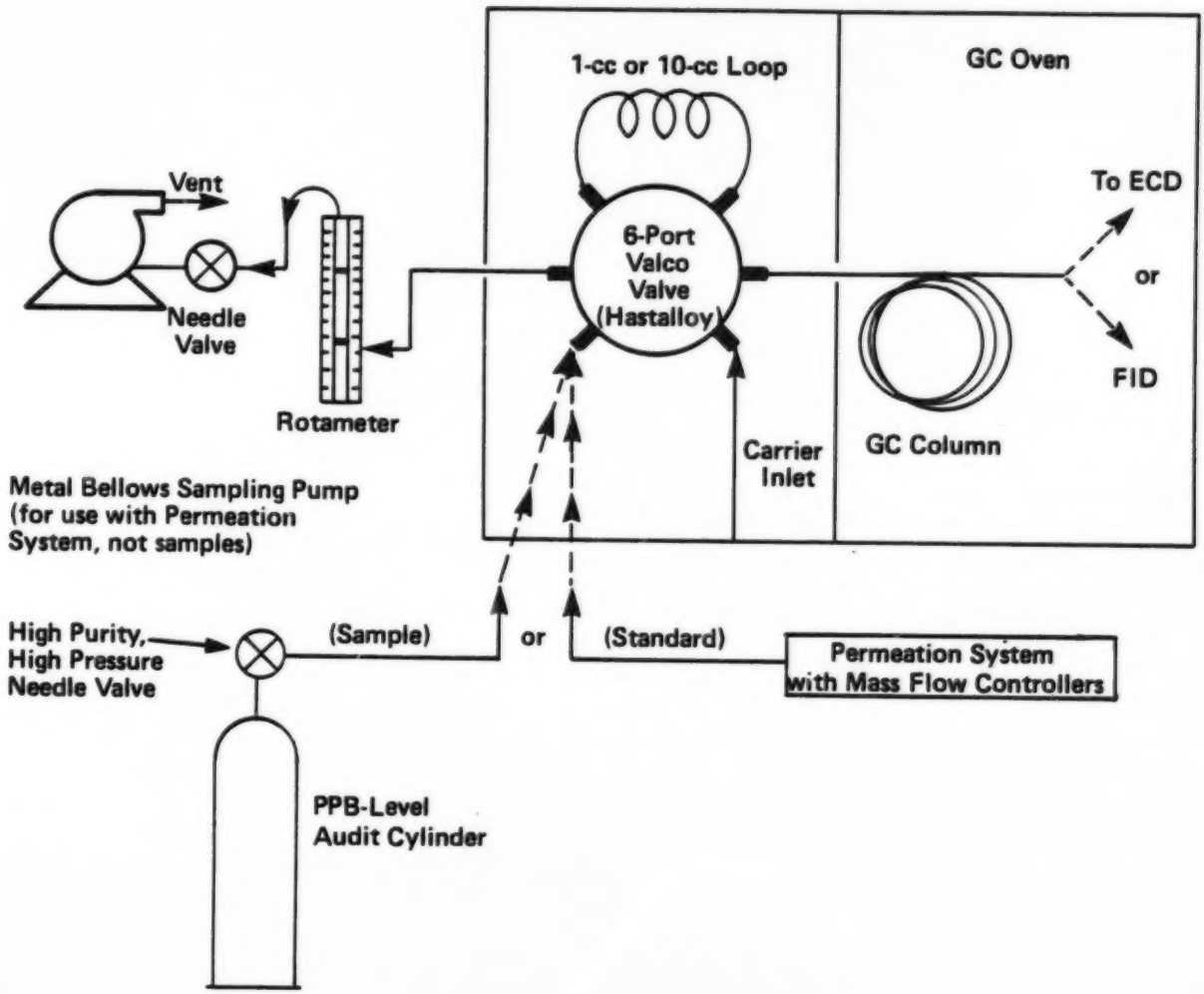

Figure 1. Schematic of the Gas Chromatographic System

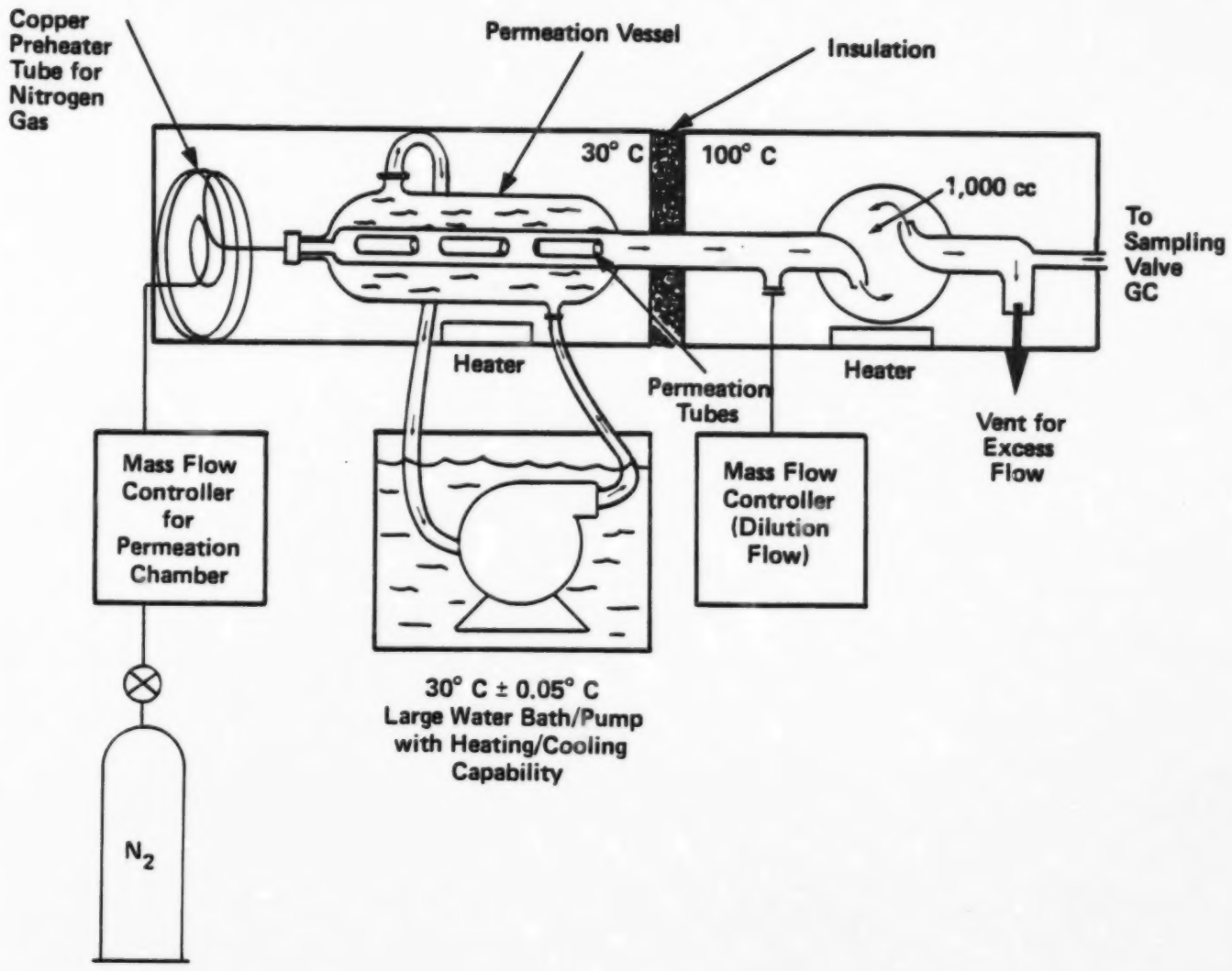

Figure 2. Schematic of the Permeation System

the column. The first injection of a sample or standard is not considered quantitative, but instead is used to condition stainless steel lines and valves. Triplicate injections of the sample are always made to determine the measurement precision.

Quality Control Procedures

Certain quality control procedures are followed, for example, equilibrating the permeation system before taking an aliquot for GC measurement. A set of primary standards for Group I and Group II multicomponent mixtures are analyzed each day cylinder gases are analyzed as an internal quality control to verify the constancy of the detector response. The primary standards were prepared and analyzed by the National Bureau of Standards (NBS) as part of an Interagency Agreement with EPA. Generally, RTI analysis results have been in close agreement with the NBS analysis values. If results vary by more than 10 percent, a calibration error is assumed and the permeation system, and sample valves are checked for leaks under both positive and negative pressure. Leak detection fluid is used to check for leaks under positive pressure. Freon is used to check for leaks under negative pressure. This is done by monitoring the freon peak on the GC. Once all of the fittings are leak tight, a recalibration of GC is performed. Blank measurements are taken during cylinder analysis. Blank measurements have shown no signal above the baseline.

Results and Discussion

Stability Studies

The data collected over time from the measurement of cylinder concentrations were used to estimate the stabilities of the repository gases. Cylinder gas stability data are important for several reasons. First, the gas mixture must be stable to be considered by EPA to be a reliable audit material; second, if shown to be stable, these cylinder gases can be used by other investigators as calibration standards and/or quality control check samples; finally, future EPA regulations could include performance audits as a means of assessing the accuracy of the measurement systems.

The term "stability," as it pertains to this study of gaseous compounds, is defined as the absence of change in concentrations over time for a given cylinder at a specified concentration level within the reported 95 percent uncertainty of this level. In this project, the gas mixtures were analyzed twice by the manufacturer before shipment to RTI. These analyses were performed to demonstrate that the components in the cylinder were stable at the manufacturer facility before shipment to RTI. The gas mixtures are then analyzed at RTI upon receipt from the specialty gas vendor to corroborate the vendor's analysis. The gas mixtures are again analyzed at 2 months, 6 months and at one year following the initial RTI analysis to determine the stability of the gas mixtures. Also, accuracies of the cylinder gas analyses for all compounds have been determined using the NBS primary standards on each day of RTI analysis. As an example, the stability data for one cylinder of Group I compounds is shown in Table III. An examination of the stability data for all Group I cylinders usually vary by less than 10 percent for 4 analyses over a 12 month period. Since only 6 months of

Table III. Stability study for Group I compounds
(Cylinder I-A2)

ACTIVITY	COMPOUND				
	Carbon tetra-chloride	Chloroform	Perchloro-ethylene	Benzene	Vinyl Chloride
MANUFACTURER ANALYSIS					
Theoretical PPB	(9.5)	(39.6)	(9.4)	(18.9)	(20.0)
1st Analysis Date	11/17/83	11/17/83	11/17/83	12/06/83	12/06/83
PPB	(7.7)	(42.1)	(9.2)	(15.8)	(18.3)
2nd Analysis Date	12/16/83	12/16/83	12/16/83	12/15/83	12/15/83
PPB	(7.1)	(41.2)	(7.7)	(19.6)	(19.2)
RTI ANALYSIS					
1st RTI Analysis Date	1/06/84	1/06/84	1/06/84	1/23/84	1/23/84
PPB	(9.3)	(40.0)	(9.4)	(19.6)	(20.2)
2nd RTI Analysis Day*	59	59	59	49	49
PPB	(9.3)	(38.7)	(10.3)	(20.0)	(19.0)
3rd RTI Analysis Day	238	238	238	226	226
PPB	(9.5)	(40.6)	(9.5)	(19.4)	(20.6)
4th RTI Analysis Day	404	404	404	392	392
PPB	(9.9)	(37.1)	(9.1)	(19.4)	(20.4)
RTI QUALITY CONTROL**					
1st RTI Analysis Date	1/06/84	1/06/84	1/06/84	1/23/84	1/23/84
PPB	(20.6)	(22.8)	(31.7)	(17.5)	(14.3)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)
2nd RTI Analysis Day	59	59	59	49	49
PPB	(20.3)	(8.9)	(23.6)	(18.6)	(15.0)
NBS Concentration PPB	(19 ± 1)	(9 ± 1)	(22 ± 1)	(17 ± 1)	(15 ± 1)
3rd RTI Analysis Day	238	238	238	226	226
PPB	(21.1)	(24.3)	(29.5)	(19.0)	(15.3)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)
4th RTI Analysis Day	404	404	404	392	392
PPB	(20.6)	(23.2)	(28.7)	(18.1)	(14.1)
NBS Concentration PPB	(21 ± 1)	(23 ± 1)	(29 ± 2)	(17 ± 1)	(15 ± 1)

*Number of days after 1st RTI analysis date.

**NBS primary standard was analyzed as internal quality control on each day of RTI analysis. A different NBS primary standard was used on the 2nd RTI analysis. RTI analysis concentrations are based on permeation tube standards.

stability analyses are currently available on Group II cylinders, a statement on any significant change will be made in a future report. A significant change indicates changes in cylinder contents (i.e., instability) and the imprecision of the measurement process. The possible sources of experimental error that could contribute to measurement imprecision include: (1) the variability of the sampling and injection technique used for analyses; (2) stability of and/or accuracy of calibration standards; and (3) the accuracy of reproducing permeation tube standards. Each of these sources of variability contributes to the net uncertainty of the resulting data presented in Table III. Estimates of day-to-day net uncertainty (repeatability) for Group I compounds are discussed below. The repeatability measurements to determine the total uncertainty for Group II compounds have not been completed and will be shown in a future report.

Uncertainty Determination

To assess the accuracy of concentration values assigned to each organic in the low concentration Group I cylinders, the individual uncertainties associated with the various steps in the standardization and measurement procedure must be identified and quantified. There are at least five principal sources of error in the standardization and measurement process which include: (1) the uncertainty in the permeation rate due to gravimetric weight loss measurement; (2) the uncertainty in the calibration of the mass flow controller; (3) the uncertainty in the temperature of the dilution chamber which effects the permeation rate; (4) the uncertainty in the injection and measurement of permeation standards; and (5) the uncertainty in the injection and measurement of samples. The procedures used to determine the uncertainties are given below. The procedures used for the first three variables were similar to those used by NBS.[1]

Permeation Rate Uncertainty. Permeation tubes containing five organics were evaluated for effusion rate stability at constant temperature. The uncertainty in the permeation rate depends principally on two variables which include the temperature of the dilution chamber and the gravimetric weight loss measurement. The estimated uncertainties for each variable is shown in Table IV.

As shown in Table IV, the uncertainty of permeation rate for gravimetric determination varied from 0.1-1.9% for the five compounds. The uncertainty of permeation rate due to the temperature fluctuations in the dilution chamber (30° ± 0.1°C) was calculated to be 2% at 95 percent confidence from the following equation:

$$\log P_1 = \log P_0 + \alpha (T_1 - T_0) \qquad (1)$$

Where:

T_0 = The device certification temperature (30.0°C)

T_1 = Change in temperature (30.1°C)

P_0 = Permeation rate at T_0°C

P_1 = Permeation rate at T_1°C

Table IV. Results of undertainties for each organic in Group I cylinders (percent coefficient of variation)*

Variable	Carbon tetra-chloride	Chloro-form	Perchloro-ethylene	Vinyl Chloride	Benzene
Permeation Rate (gravi-metric determination)	1.9	0.1	0.3	0.7	0.6
Permeation Rate (temper-ature effect)**	2.0	2.0	2.0	2.0	2.0
Injection of Standards	0.9	0.2	0.3	1.3	1.7
Flow-Rate of Standards	0.3	0.3	0.3	0.3	0.3
Injection of Samples	0.8	0.4	1.2	1.9	1.7
Total Uncertainty	3.0	2.1	2.4	3.2	3.2

*At 95% confidence limit.

**Based on theoretical calculation.

α = A function of permeant gas and permeable material (manufacturer recommended mean value of 0.034).

The estimated uncertainty of the permeation rate due to the above two variables was then calculated from the expression:

$$S_{\text{Permeation rate, est.}} = 2\sqrt{CV_1^2 + CV_2^2} \qquad (2)$$

Where:

S = Net uncertainty of the permeation rate.

CV_1 = Percent coefficient of variation of permeation rate due to weight loss.

CV_2 = Percent coefficient of variation of permeation rate due to temperature effect.

2 = Two standard deviations.

Typical estimated uncertainties for the permeation rate determination ranged from 2.0 - 3.0% (percent coefficient of variation) for the five organics at 95% confidence limits.

Precision of Calibration Standards Analysis. The uncertainty of the permeation standards analysis depends principally on two variables: the measurement of nitrogen flow passed through the chamber holding the permeation tubes and the injection of the resulting organic-nitrogen mixture into the gas chromatograph using the gas sampling valve. These two uncertainties were determined by the measurement of flow rates on each day of analysis and the triplicate injection of calibration standards into the GC (see Table IV). These measurements were made over 3 separate days. Typical estimated uncertainties for the calibration standards analysis ranged from 0.2 - 1.7% (coefficient of variation) at 95% confidence limits.

Precision of Sample Analysis. The uncertainty of the sample analysis was determined by the triplicate injection of gas samples from three low ppb audit cylinders (5 - 50 ppb levels) into the gas chromatograph using the automatic gas sampling valve over 3 separate days. The imprecision for repeated analysis of the organics ranged from 0.4 to 1.9% (coefficient of variation), reflecting the precision of mean concentrations determined by analysis.

Total Uncertainty Determination. The estimates of total relative uncertainty for each of 5 organics were calculated at the 95% confidence limit by substitution of the percent coefficient of variation for each variable into the equation:

$$U_T = 2\sqrt{\left(CV_1^2 + CV_2^2 + CV_3^2 + CV_4^2 + CV_5^2\right)} \qquad (3)$$

Where:

U_T = Total uncertainty of all 5 variables (percent coefficient variation).

CV_1 = Percent coefficient of variation of permeation rate due to weight loss measurement.

CV_2 = Percent coefficient of variation of permeation rate due to temperature effect.

CV_3 = Percent coefficient of variation for injection of standards.

CV_4 = Percent coefficient of variation of flow-rate measurements.

CV_5 = Percent coefficient of variation for injection of samples.

The estimated uncertainty for all 5 organics ranged from 2.1 - 3.2% (coefficient of variation) at 95% confidence limits.

Performance Audits

As stated previously, the USEPA (through RTI) supplies repository cylinders for audits upon request from the EPA, State or Local agencies or their contractors. A contractor must be performing hazardous waste trial burn tests at the request of EPA, a state or local agency in order to qualify for the performance audit. Performance audits prior to the actual trial burn tests to assess proficiency in conducting a trial burn test are also encouraged.

When a request is received for an audit, the cylinder pressures are measured and the cylinder and a briefcase containing a glass manifold and necessary equipment to deliver the audit gas from the audit cylinder are shipped by overland carrier to the audit site. General instructions for conducting the audit are included with the cylinder. The audit results are reported to the Agency (EPA, State or Local) Coordinator requesting the audit. There is no charge for the audit except the cost of returning the audit cylinder.

To date, 28 performance audits have been initiated; 20 are complete. Representative audit results are presented in Table V. The sampling and analysis methods used by auditees during performance audits and the results for all the analyses conducted to date are presented elsewhere.[2] The audit results are usually within $\pm$ 50 percent of the audit concentrations measured by RTI. The principal results of these audits have been to assure all concerned that measurements are being performed properly, to uncover problems that could be corrected, and to document the accuracy of the measurement systems used during the hazardous waste trial burn tests.

Summary and Conclusions

A repository of 14 gaseous organic compounds at parts-per-billion levels in compressed gas cylinders has been used successfully in audits to assess the relative accuracy of analytical systems, especially those used during hazardous waste trial burn tests. The repository consists of two mixtures of five and nine organic compounds each. These mixtures were blended in aluminum cylinders in a balance gas of nitrogen. The five compo-

Table V. Example of performance audit results

Audit No.	Use of Audit Material	Audit Material	RTI Measured Conc. (ppb)	Audit Result Bias, %[a]
5	VOST method evaluation prior to trial burn	Carbon tetrachloride	11.0	+31
		Chloroform	45.0	+20
		Vinyl chloride	20.5	+7
11	VOST method evaluation prior to trial burn	Carbon tetrachloride	11.0	+5
		Chloroform	47.4	+3
		Perchloroethylene	11.3	+5
		Benzene	25.2	-7
		Vinyl chloride	19.8	-85
12	VOST method evaluation prior to trial burn	Carbon tetrachloride	6.6	+21
		Chloroform	7.4	+12
		Perchloroethylene	21.9	+56
		Benzene	26.0	+47
		Vinyl chloride	29.6	-52
17	VOST method evaluation prior to trial burn	Carbon tetrachloride	9.9	-19
		Chloroform	42.0	-32
		Perchloroethylene	9.6	-14
		Benzene	20.0	-21
		Vinyl chloride	19.8	-41

[a]Audit Result Bias = $100 \times \frac{\text{Client Measured Conc.} - \text{RTI Measured Conc.}}{\text{RTI Measured Conc.}}$

nent mixture (Group I) contains carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene. The nine component mixture (Group II) includes trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane, dichlorodifluoromethane, bromomethane, methyl ethyl ketone, and 1,1,1-trichloroethane. Accuracies of the cylinder gas analyses for Group I and Group II compounds have been determined using the NBS primary standards. In addition, the EPA is now in the process of preparing gas cylinders containing eight other organic compounds (Group III) at low parts-per-billion levels (15 to 150 ppb) for performance audits. These eight organic compounds are: pyridine, vinelidiene chloride, 1,1,2-trichloro 1,2,2-trifluoroethane (Freon 113), 1,2-dichloro 1,1,2,2-tetrafluoroethane (Freon 114), acetone, 1,4- dioxane, toluene and chlorobenzene. However, these cylinders will not be available for audits until stability of these components have been established. The audit cylinders will become available on or after September 1985.

The stability data obtained to date for the audit mixtures of five and nine organic compounds indicate that the mixtures are adequately stable for use in performance audits during hazardous waste trial burn tests. Estimates of total uncertainty (repeatability) for Group I compounds have been determined to be in the range of 2.1 - 3.2% (coefficient of variation) at 95% confidence limits. The estimate of total uncertainty (repeatability) for Group II compounds is in progress at the present time and will be presented in a future publication.

The compressed cylinder gases containing selected organics can be used successfully for performance audits to assess and document the precision and accuracy of the analytical measurements. Hence, it is recommended that a performance audit using EPA audit cylinders be conducted during each hazardous waste trial burn test as a routine quality assurance procedure. Any federal, state or local agency or its contractor planning hazardous waste trial burn tests may request a performance audit by contacting Mr. Robert Lampe, USEPA, Environmental Monitoring Systems Laboratory, Quality Assurance Division, Research Triangle Park, North Carolina 27711.

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